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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
09/895,517	06/28/2001	Ralf Wolleschensky	GK-ZEI-3099/500343.20099	8222

7590 12/31/2003

REED SMITH LLP
375 Park Avenue
New York, NY 10152

EXAMINER	
PRITCHETT, JOSHUA L	
ART UNIT	**PAPER NUMBER**
2872	

DATE MAILED: 12/31/2003

Please find below and/or attached an Office communication concerning this application or proceeding.

Office Action Summary	Application No. 09/895,517	Applicant(s) WOLLESCHENSKY ET AL.	
	Examiner Joshua L Pritchett	Art Unit 2872	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondenc address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☒ Responsive to communication(s) filed on 04 November 2003.

2a)☐ This action is **FINAL**. 2b)☒ This action is non-final.

3)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

4)☒ Claim(s) 1-90 is/are pending in the application.

4a) Of the above claim(s) _____ is/are withdrawn from consideration.

5)☐ Claim(s) _____ is/are allowed.

6)☒ Claim(s) 1-90 is/are rejected.

7)☐ Claim(s) _____ is/are objected to.

8)☐ Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

9)☐ The specification is objected to by the Examiner.

10)☒ The drawing(s) filed on 28 June 2001 is/are: a)☒ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

11)☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. §§ 119 and 120

12)☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

a)☒ All b)☐ Some * c)☐ None of:

1.☒ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

13)☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. § 119(e) (to a provisional application) since a specific reference was included in the first sentence of the specification or in an Application Data Sheet. 37 CFR 1.78.

a) ☐ The translation of the foreign language provisional application has been received.

14)☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. §§ 120 and/or 121 since a specific reference was included in the first sentence of the specification or in an Application Data Sheet. 37 CFR 1.78.

Attachment(s)

1) ☒ Notice of References Cited (PTO-892)
2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
3) ☐ Information Disclosure Statement(s) (PTO-1449) Paper No(s) _____ .
4) ☐ Interview Summary (PTO-413) Paper No(s). _____ .
5) ☐ Notice of Informal Patent Application (PTO-152)
6) ☐ Other:

DETAILED ACTION

This action is in response to the Request for Continued Examination filed November 4, 2003. Claims 1-8 and 48 have been added and claims 87-90 have been added as requested by applicant.

Claim Rejections - 35 USC § 112

The following is a quotation of the second paragraph of 35 U.S.C. 112:

> The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

Regarding claims 1-47 and 87-89, the phrase "such as" renders the claim indefinite because it is unclear whether the limitations following the phrase are part of the claimed invention. See MPEP § 2173.05(d).

Claims 48-86 and 90 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. Regarding claims 48 and 90, the claim language fails to clearly define the limitations of the claim as to which type of wavelength dependent behavior is examined by the claimed method. The remaining claims depend from claim 48 and inherit the deficiencies thereof.

Claim Rejections - 35 USC § 102

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

> A person shall be entitled to a patent unless –
>
> (b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

Claims 1, 7, 20, 21, 24, 25, 48, 52, 62, 63, 66 and 67 are rejected under 35 U.S.C. 102(b) as being anticipated by Jeffers (US 5,486,915).

Regarding claims 1 and 48, Jeffers discloses a method for optical detection of characteristic quantities of the wavelength-dependent behavior (abstract lines 2-3) of an illuminated specimen (abstract lines 1-2), such as the emission behavior or absorption behavior, preferably the fluorescence (abstract lines 4-5) or luminescence or phosphorescence or enzyme-active light emission or enzyme-active fluorescence by determining at least one centroid (abstract lines 7-8). Jeffers discloses in the abstract that using a "selected wavelength" of light excites the sample, in this case wood pulp. Jeffers further discloses that the "fluorescence emission" from the sample is determined and the concentration of a particular compound in the sample is calculated based on either the centroid method or a band ratio method.

Regarding claims 7 and 52, Jeffers further teaches spectral weighting carried out between a plurality of detection channels, summing of the weighted channels of the signals of the detection and summing the detection channels (col. 5 lines 55; Eq. 2).

Regarding claims 20 and 62, Jeffers further teaches the use of color-coded fluorescence imaging (col. 3 line 67).

Regarding claims 21 and 63, Jeffers further teaches the superposition of additional images (Fig. 1). Fig. 1 shows several graph lines superimposed onto one set of axes.

Regarding claims 24 and 66, Jeffers further teaches a comparison of the measured signal to a reference carried out by comparators in detection channels and in case the reference signal is not reached or is exceeded a change in the operating mode of the detection channel occurs (140). Element 140 of Jeffers is a control element which inherently receives a detection signal and compares it to a reference signal to determine how to modify the production process.

Regarding claims 25 and 67, Jeffers teaches that respective detection channel is switched off or not taken into account (140). Element 140 of Jeffers is a control element which inherently shuts off control when the reference signal is matched by the detection signal within acceptable error.

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

> (a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

Claims 2-3, 10, 14, 26, 32, 42, 45, 55, 66, 74 and 83 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Yang.

Regarding claims 2 and 3, Jeffers teaches the invention as claimed including using the centroid to determine the concentration of a component of a composition but lacks specific reference to the use of the centroid detection to determine the amount of dye in a composition. Yang teaches the emission radiation of fluorochromes is carried out for distinguishing different dyes and/or determine the local dye composition of an image point when a plurality of dyes are used simultaneously or determine the local shift of the emission spectrum depending on the local environment to which the dye or dyes is or are attached and/or for measuring emission ratio dyes for determining ion concentration (col. 3 lines 32-35). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention determine the dye concentration of a composition as taught by Yang for the purpose of determining if any hazardous components existed within the tested composition.

Regarding claims 10 and 55, Jeffers teaches the invention as claimed but lacks reference to converting the signal to a digital signal. Yang teaches signal detection conversion to digital and reading out of the converted signal and the weighting and summing carried out digitally in a computer (col. 6 lines 15-23, col. 17 lines 26-28). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention convert the signal into a digital signal as taught by Yang for the purpose of more easily viewing the collected results.

Regarding claim 14, Jeffers teaches the invention as claimed but lacks reference to nonlinear distortion of the signal. Yang teaches the signals of the detector channels are

influenced by nonlinear distortion of the input signals (col. 26 line 1). In this case experimental noise is taken to mean the same thing as nonlinear distortion. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers signal influenced by nonlinear distortion as taught by Yang of the purpose of accounting for experimental noise in the collected results.

Regarding claims 26 and 68, Jeffers teaches the invention as claimed but lacks reference to narrowing the spectral region. Yang teaches the relevant spectral region is narrowed (col. 25 lines 7-8). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention narrow the spectral region as taught by Yang for the purpose of targeting a specific material or wavelength during process monitoring.

Regarding claims 32 and 74, Jeffers teaches the invention as claimed but lacks a microscope. Yang teaches the use of the spectral detection in a microscope (col. 25 line 7). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention include a microscope as taught by Yang for the purpose of viewing small variations in the emission pattern of the tested sample.

Regarding claims 42, 45 and 83, Jeffers teaches the invention as claimed but lacks reference to brightfield imaging. Yang teaches the use of brightfield imaging (col. 6 lines 5-6). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention use brightfield imaging for the purpose of easily viewing the results from the collected data.

Regarding claims 43 and 84, Jeffers teaches the invention as claimed but lacks reference to point imaging. Yang teaches the use of point imaging (col. 3 line 39). It would have been

obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention use point imaging as taught by Yang for the purpose of viewing small variations in the emission pattern of the tested sample.

Claims 4-6, 8, 9, 13, 15, 17-19, 49-51, 53, 54 and 58-61are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Okubo.

Regarding claims 4-6 and 49-51, Jeffers teaches the invention as claimed, but lacks reference to the splitting of the emission radiation by a dispersive element. Okubo teaches the use of a dispersive element to split the emission radiation for the sample being examined (page 10 lines 31-33). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a dispersive element to split the emission radiation entering the Jeffers invention for the purpose of sending the signal to several different detectors for data processing and analysis.

Regarding claims 8, 9, 13, 53, 54, and 58, Jeffers lacks the use of a weighting curve in the analysis of the data collected. Okubo teaches the use of both straight line and adjustable weighting curve during data analysis (page 7 lines 19-34). In this case the equations provided by Okubo are taken to be numerical representations of the equations that determine the weighting curve. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a weighting curve to analyze the data collected by the Jeffers invention for the purpose of quicker and more precise calculations through the use of a reference material.

Regarding claim 15 and 16, Jeffers further lacks the influence of experimental noise on integration parameters and amplification during the data analysis process. Okubo teaches that the integration parameters and amplification are adjustable based on the signal input (page 7 lines 19-34). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to make the integration parameters and the amplification of the signal adjustable for the purpose of collecting more precise results from the experimentation through the elimination of experimental noise.

Regarding claims 17 and 59, Jeffers does teach the conversion of the signal from analog to digital (col. 25 lines 35-36).

Regarding claims 18 and 60, Jeffers further lacks the weighting of the signals from the individual channels through the use of weight curves. Okubo teaches the use of weight curves and the application of weight curves in summing the results from different channels (page 7 lines 19-34). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a weighting curve to analyze the data collected by the Jeffers invention for the purpose of more precise and reliable calculations.

Regarding claims 19 and 61, Jeffers also teaches the sum signal generating an image (col. 25 lines 29-30).

Claims 11, 12, 27, 56, 57, and 69 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Lee.

Regarding claims 11 and 56 Jeffers teaches the invention as claimed but lacks the dimension of analog processing through the use of resistance cascade. Lee teaches the use of

resistors in combination as a means of processing the collected data (col. 10 lines 28-34). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use resistors in combination as a data processing means within the Jeffers invention for the purpose of sorting the intensity of the light emission by the amount of electricity created by the photons contacting the detector.

Regarding claims 12 and 57 Jeffers further lacks adjustable resistance within the resistors used to process the data collected. Lee teaches the use of adjustable resistors (col. 10 line 34). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use adjustable resistors for the purpose of increased flexibility in the application of the Jeffers invention.

Regarding claims 27 and 69 Jeffers further lacks signals of detection channels being generated by an integrator circuit. Lee teaches the use of an integration circuit to generate the signal of a detection channel (col. 10 lines 28-34). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use an integrator circuit in the generation of the signals from the Jeffers invention for the purpose of quick and reliable conversion of the light intensity to an electronic signal usable by a computer.

Claims 22, 23, 64 and 65 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Okubo as applied to claims 8 and 53 above, and further in view of Hochman.

Jeffers in view of Okubo teaches the invention as claimed but lacks the use of either a weighting curve or a lookup table. Hochman teaches the use of a lookup table for use in combination with position and sum signals as well as in the representation of different dyes

and/or the spread of the generated image (col. 12 lines 12-15). It would have been obvious to a person of ordinary art at the time the invention was made to use a lookup table as described in Hochman in combination with the Jeffers in view of Okubo invention for the purpose of making the generated images easier to understand and to increase the speed of data analysis.

Claims 28-30, 34, 70-72, and 76 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Kash.

Regarding claims 28 and 70 Jeffers teaches the invention as claimed but lacks a specific reference to photon counting. Kash teaches the use of photon counting as a means to determine the intensity of light emitted from a sample (col. 7 lines 46-48). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a photon detector as a means of collecting the emission intensity for the Jeffers invention because the use of a photon detector is commonly known in the art.

Regarding claims 29 and 71 Jeffers further lacks photon counting carried out in a time relation. Jeffers does teach a time dependent relationship within the invention (col. 5 lines 36-37). Kash teaches the use of time related photon counting (col. 3 lines 5-20). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use time related photon counting in the Jeffers invention for the purpose of measuring how the emissions of the sample change with respect to time.

Regarding claims 30, 34, 72, and 76, Jeffers further lacks the specific mention of the detection of single photons or multiphoton fluorescence. Kash teaches the detection of multiphoton fluorescence (col. 6 lines 47-48). It would have been obvious to a person of

ordinary skill in the art at the time the invention was made to use single or multiphoton fluorescence detection within the Jeffers invention for the purpose of collecting data related to the intensity of the emission of the sample being examined.

Claims 35, 39, 41, 44, 46, 77, 81, 82, 85, and 86 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Simon.

Regarding claims 25, 39, 46, 77, 81 and 86, Jeffers teaches the invention as claimed but lacks reference to either confocal or nonconfocal detection. Simon teaches the use of both confocal and nonconfocal detection (col. 4 line 2). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use either confocal or nonconfocal detection in combination with the Jeffers invention for the purpose of focusing the emission of the sample to the appropriate location of a detector.

Regarding claims 41, 44, 82 and 85, Jeffers further lacks reference to either descanning or nondescanning detection. Simon teaches the use of either descanning or nondescanning detection (col. 3 lines 12-13). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use either descanning or nondescanning detection within the Jeffers invention for the purpose of determining the emission of the sample being tested.

Claims 33, 36, 37, 38, 40, 47, 75, 78, 79 and 80 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Yagi.

Regarding claims 33 and 75, Jeffers teaches the invention as claimed but lacks reference as to the method of scanning the sample during data collection. Yagi teaches a scanning

microscope (col. 2 lines 6-7). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a scanning microscope within the Jeffers invention for the purpose of collecting data from a wide spatial range within the test sample and to allow the ability to pinpoint a specific location within the sample.

Regarding claims 36, 40 and 78, Jeffers further lacks a scanning arrangement for the microscope. Yagi teaches a scanning arrangement (col. 4 lines 3-4). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to provide the Jeffers invention with a scanning arrangement for the purpose of collecting data from a wide spatial range within the test sample and to allow the ability to pinpoint a specific location within the sample.

Regarding claims 37, 38, 47, 79, and 80, Jeffers further lacks reference to either an X-Y scanning table or illumination means for a scanning means. Yagi teaches the use of an X-Y scan table (col. 4 lines 3-4). Yagi further teaches a means of illumination for the X-Y scanning means (col. 4 lines 1-2). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to provide the Jeffers invention with an X-Y scanning table and a means of illumination for the X-Y scanning means for the purpose of collecting data from a wide spatial range within the test sample and to allow the ability to pinpoint a specific location within the sample.

Claims 31 and 73 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Tuuanen.

Jeffers teaches the invention as claimed, but lacks reference to the placement of the sample on a microtiter plate. Tuuanen teaches the placement of a samples used in fluorescence microscopy on a microtiter plate (col. 2 lines 45-47). It would have been obvious to a person of ordinary skill in the art at the time the invention was made to place the sample on a microtiter plate because this practice is widely known and used in the art.

Claims 87-90 are rejected under 35 U.S.C. 103(a) as being unpatentable over Jeffers in view of Yang and Okubo.

Jeffers discloses a method for optical detection of characteristic quantities of the wavelength-dependent behavior of an illuminated specimen, such as the emission behavior and/or absorption behavior, preferably the fluorescence and/or luminescence and/or phosphorescence and/or enzyme-active light emission and/or enzyme-active fluorescence by determining at least one centroid (abstract lines 7-8). Jeffers lacks specific reference to the use of the centroid detection to determine the amount of dye in a composition. Yang teaches the emission radiation of fluorochromes is carried out for distinguishing different dyes and/or determine the local dye composition of an image point when a plurality of dyes are used simultaneously or determine the local shift of the emission spectrum depending on the local environment to which the dye or dyes is or are attached and/or for measuring emission ratio dyes for determining ion concentration (col. 3 lines 32-35). Jeffers lacks reference to the splitting of the emission radiation by a dispersive element and the use of a weighting curve. Okubo teaches the use of a dispersive element to split the emission radiation for the sample being examined (page 10 lines 31-33). Okubo teaches the use of both straight line and adjustable weighting

curve during data analysis (page 7 lines 19-34). In this case the equations provided by Okubo are taken to be numerical representations of the equations that determine the weighting curve. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to have the Jeffers invention determine the dye concentration of a composition as taught by Yang for the purpose of determining if any hazardous components existed within the tested composition. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a dispersive element to split the emission radiation entering the Jeffers invention for the purpose of sending the signal to several different detectors for data processing and analysis. It would have been obvious to a person of ordinary skill in the art at the time the invention was made to use a weighting curve to analyze the data collected by the Jeffers invention for the purpose of quicker and more precise calculations through the use of a reference material.

Response to Arguments

Applicant's arguments, see Amendment B, filed October 3, 2003, with respect to the rejection(s)of claim(s) 1 and 48 under Yang have been fully considered and are persuasive. Therefore, the rejection has been withdrawn. However, upon further consideration, a new ground(s) of rejection is made in view of Jeffers.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Joshua L Pritchett whose telephone number is 703-305-7917. The examiner can normally be reached on Monday - Friday 7:00 - 3:30.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Drew A Dunn can be reached on 703-305-0024. The fax phone number for the organization where this application or proceeding is assigned is 703-872-9318.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-0956.

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DREW DUNN
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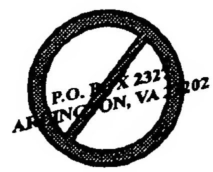

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Notic of References Cited	Application/Control No. 09/895,517	Applicant(s)/Patent Under Reexamination WOLLESCHENSKY ET AL.	
	Examiner Joshua L Pritchett	Art Unit 2872	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification
	A	US-5,486,915	01-1996	Jeffers et al.	356/318
	B	US-			
	C	US-			
	D	US-			
	E	US-			
	F	US-			
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NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
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*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

US005486915A

United States Patent [19]

Jeffers et al.

[11] **Patent Number: 5,486,915**

[45] **Date f Patent: Jan. 23, 1996**

[54] **ON-LINE MEASUREMENT OF LIGNIN IN WOOD PULP BY COLOR SHIFT OF FLUORESCENCE**

[75] Inventors: **Larry A. Jeffers**, Washington Township, Stark County; **Michael L. Malito**, Liberty Township, Trumbull County, both of Ohio

[73] Assignee: **The Babcock & Wilcox Company**, New Orleans, La.

[21] Appl. No.: **226,801**

[22] Filed: **Apr. 12, 1994**

[51] **Int. Cl.**^{6} **G01N 21/64**; D21C 7/14

[52] **U.S. Cl.** **356/318**; 250/461.1; 162/49

[58] **Field of Search** 356/317–318, 356/417; 250/458.1, 459.1, 461.1, 462.2; 162/49, 198, 263

[56] **References Cited**

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Williams, D. J., "The Application of the Ultra Violet Absorp

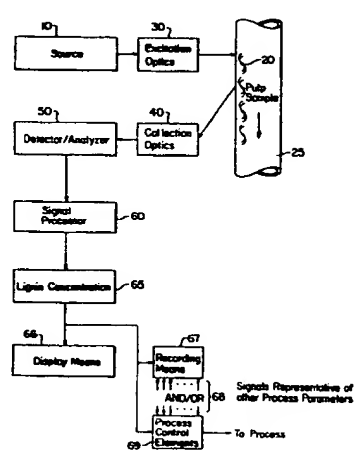
(List continued on next page.)

Primary Examiner—Vincent P. McGraw
Assistant Examiner—K. P. Hantis
Attorney, Agent, or Firm—Robert J. Edwards; Eric Marich

[57] **ABSTRACT**

Lignin concentrations from wood pulp samples are measured by applying an excitation light at a selected wavelength to the samples in order to cause the lignin to emit fluorescence. A spectral distribution of the fluorescence emission is then determined. The lignin concentration is then calculated based on the spectral distribution signal. The spectral distribution is quantified by either a wavelength centroid method or a band ratio method.

23 Claims, 6 Drawing Sheets



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BTG Sales "Kappa Number Analyzer", B 218.66 e–a Entire Paper–Product Brochure, Date Unknown.
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Kubulneiks, E. Sandstrom, P. "Control of Oxygen Delignification Based on On–Line Kappa Number Measurement" 1988 International Pulp Bleaching Conference pp. 47–51.

BTG–Product Brochure "KNA–500 Kappa Number Analyzer" Entire Paper–Date Unknown.

Horvath, J. J. & Semerjian, H. G. "Laser Excited Fluorescence Studies of Black Liquor" Proceedings of the SPIE, vol. 665, Jun., 1986, pp. 258–264.

FIG. 1

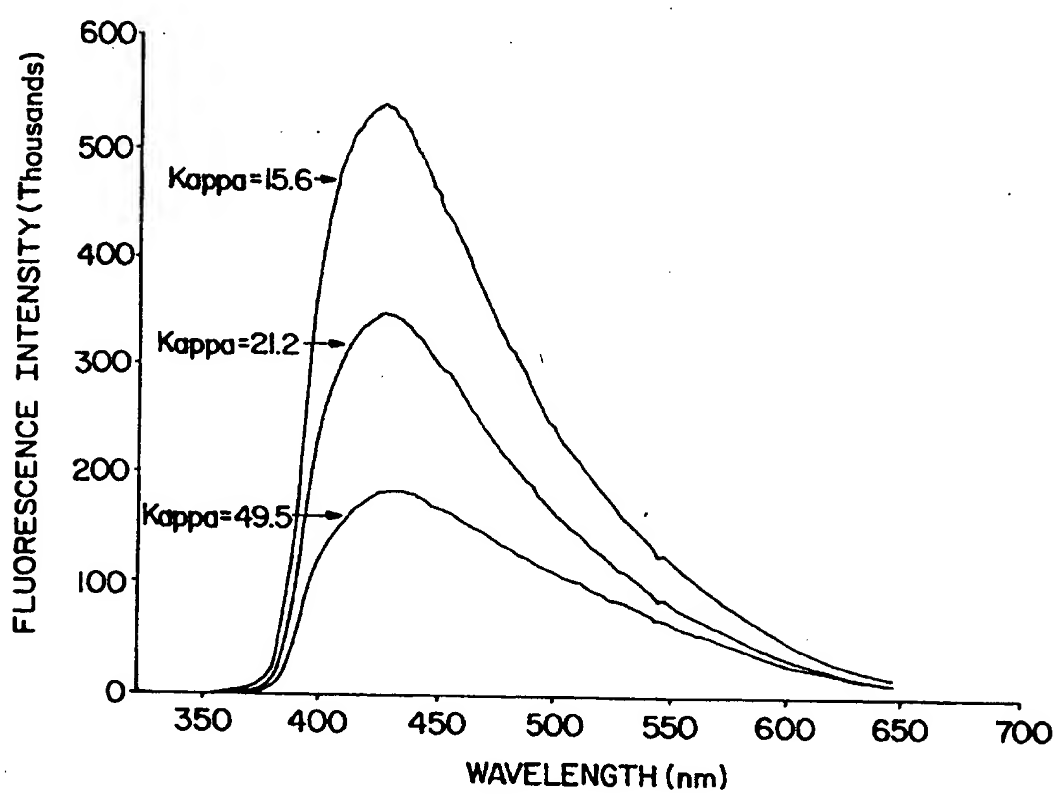

FLUORESCENCE INTENSITY (Thousands)
600
500
400
300
200
100
0
Kappa=15.6
Kappa=21.2
Kappa=49.5
350
400
450
500
550
600
650
700
WAVELENGTH (nm)

FIG. 2

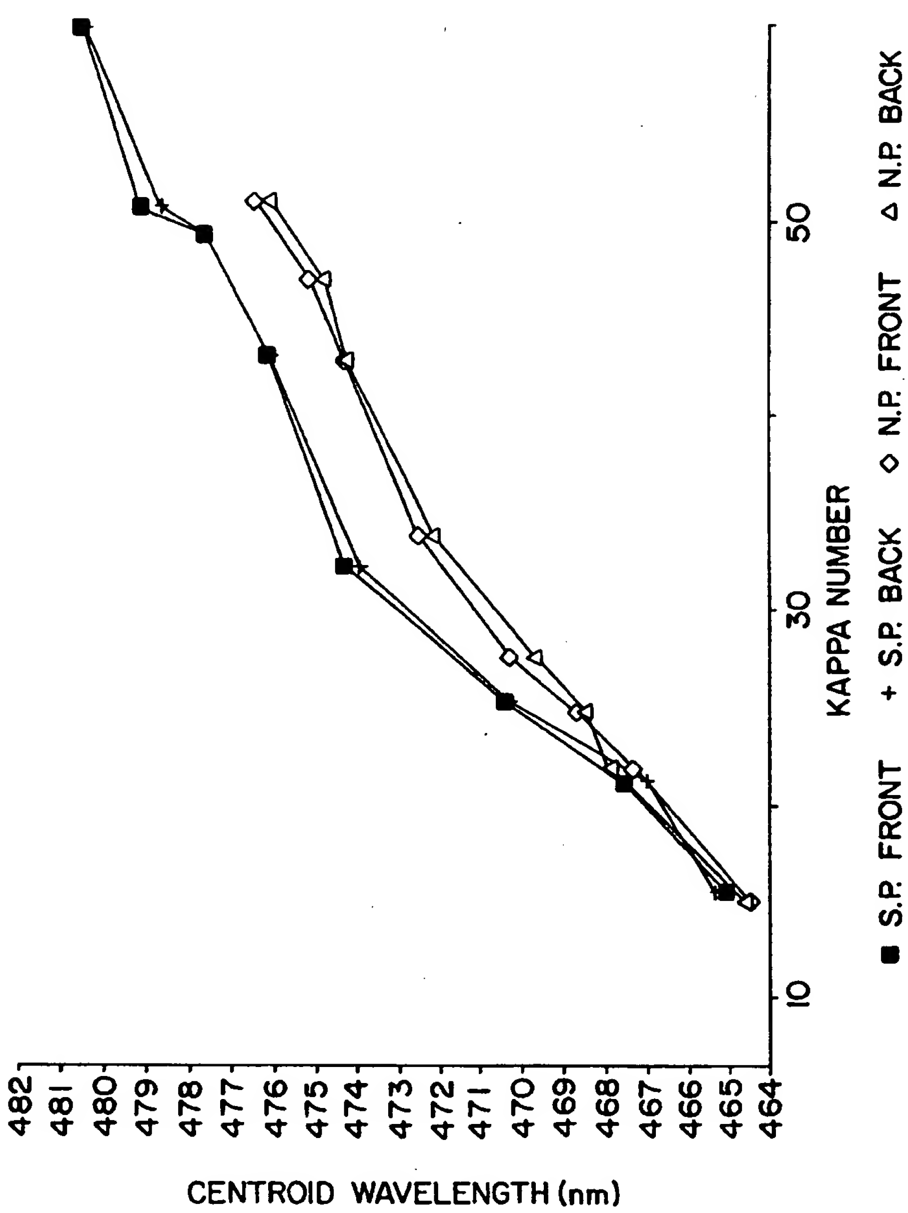

CENTROID WAVELENGTH (nm)
482
481
480
479
478
477
476
475
474
473
472
471
470
469
468
467
466
465
464
KAPPA NUMBER
10
30
50
■ S.P. FRONT + S.P. BACK ◇ N.P. FRONT △ N.P. BACK

FIG. 3

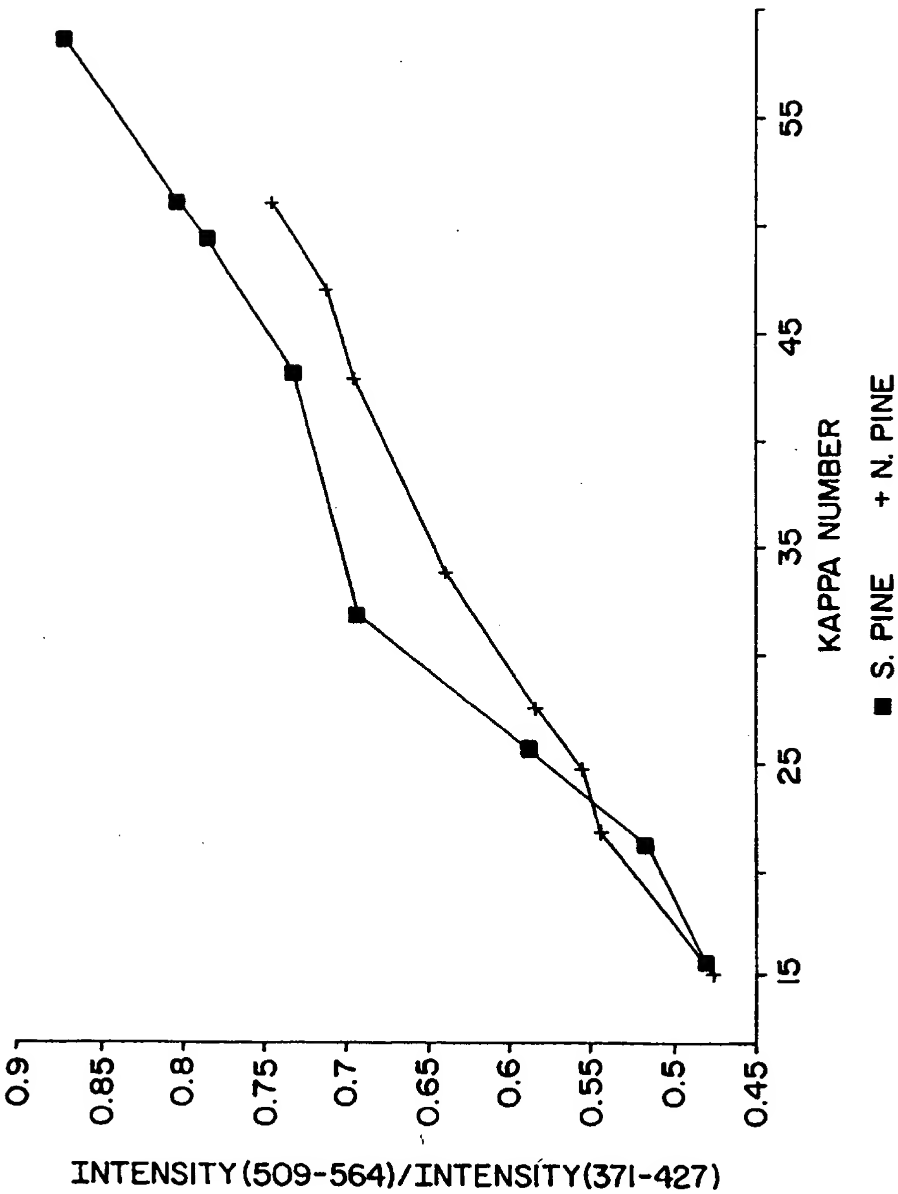

KAPPA NUMBER
15
25
35
45
55
0.9
0.85
0.8
0.75
0.7
0.65
0.6
0.55
0.5
0.45
INTENSITY(509-564)/INTENSITY(371-427)
■ S. PINE
+ N. PINE

FIG. 4

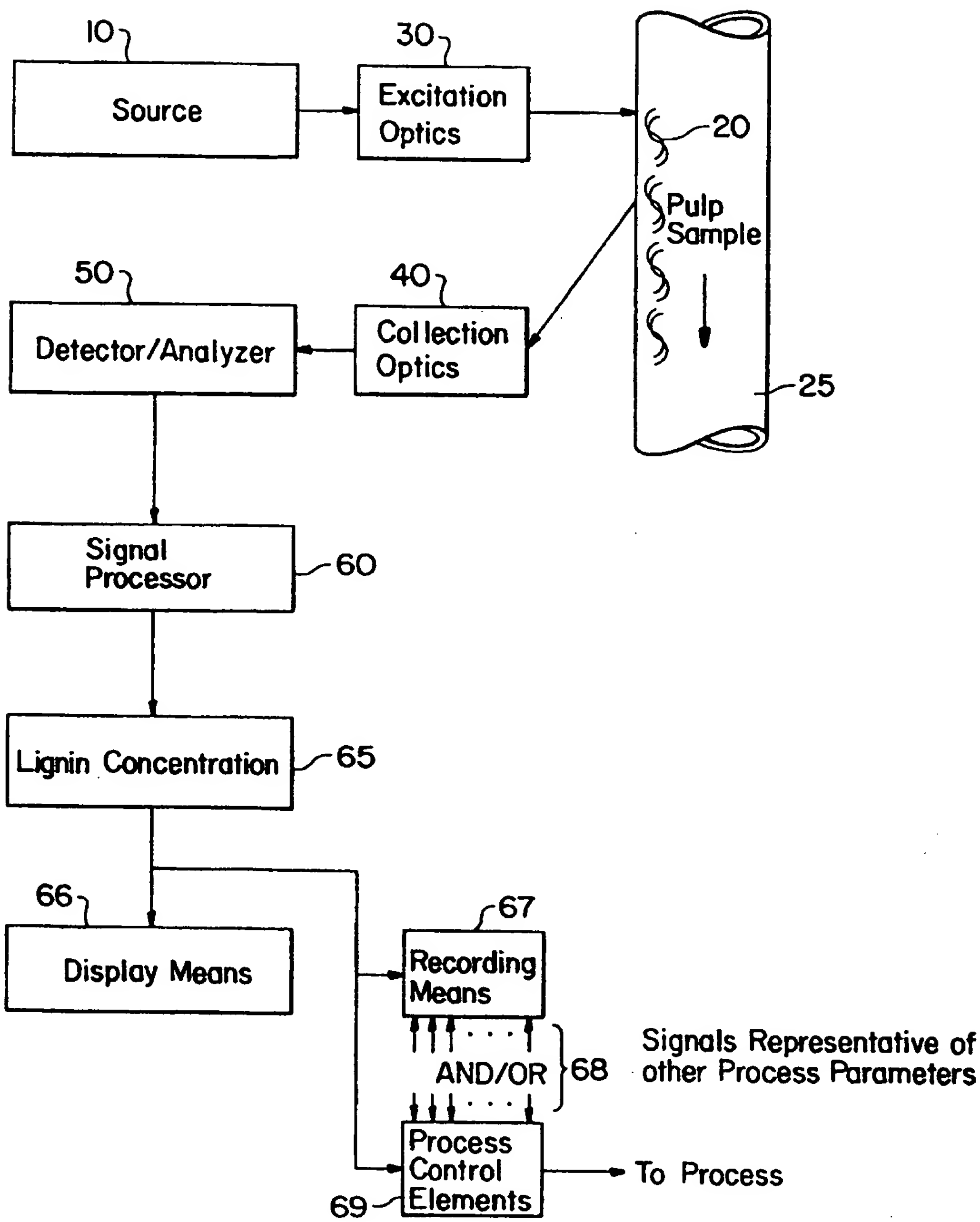

10
Source
30
Excitation Optics
20
Pulp Sample
25
50
Detector/Analyzer
40
Collection Optics
Signal Processor
60
Lignin Concentration
65
66
Display Means
67
Recording Means
AND/OR
68
Signals Representative of other Process Parameters
Process Control Elements
69
To Process

FIG. 5

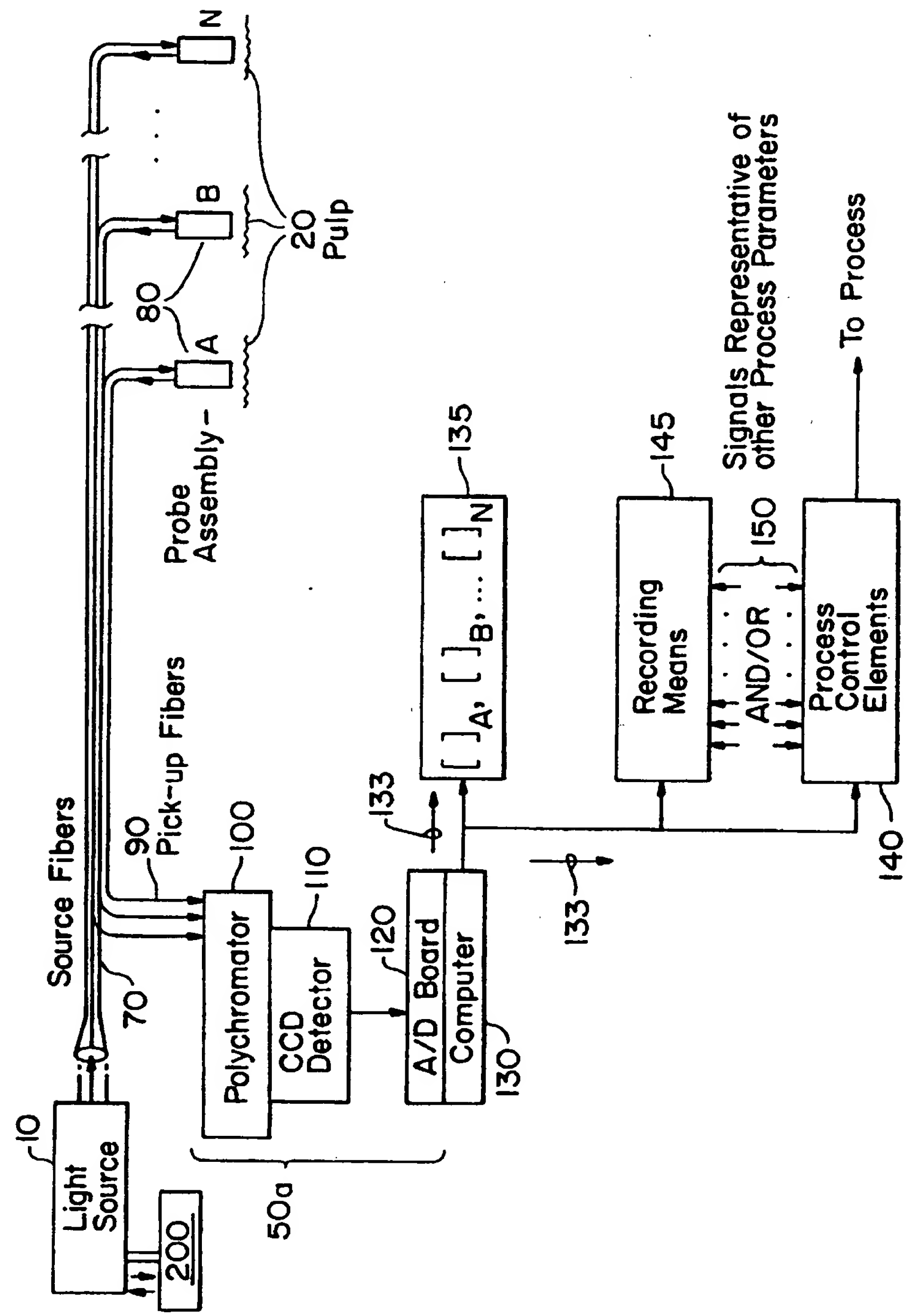
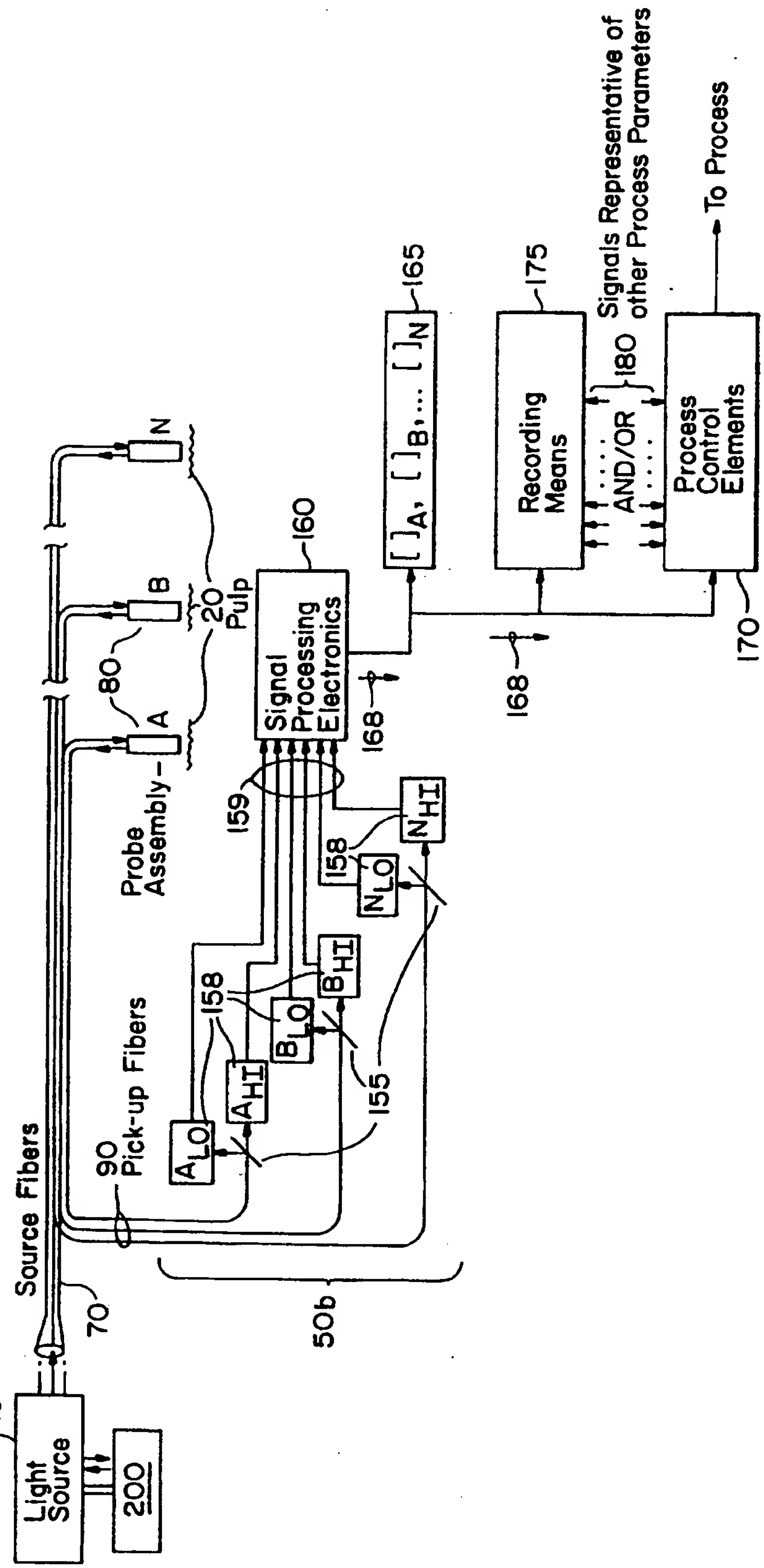

Light Source
10
200
Source Fibers
70
90
Pick-up Fibers
Probe Assembly
80
A
B
N
20
Pulp
Polychromator
100
CCD Detector
110
A/D Board
120
Computer
130
50a
133
135
[ ]A, [ ]B, ... [ ]N
Recording Means
145
150
AND/OR
Process Control Elements
140
Signals Representative of other Process Parameters
To Process

FIG. 6



Light Source
10
200
70
Source Fibers
90
Pick-up Fibers
Probe Assembly
80
A
B
N
20
Pulp
50b
155
158
159
A_{LO}
A_{HI}
B_{LO}
B_{HI}
N_{LO}
N_{HI}
Signal Processing Electronics
160
168
[ ]A, [ ]B,... [ ]N
165
Recording Means
175
AND/OR
180
Process Control Elements
170
Signals Representative of other Process Parameters
To Process

ON-LINE MEASUREMENT OF LIGNIN IN WOOD PULP BY COLOR SHIFT OF FLUORESCENCE

The Government of the United States of America has certain rights in the invention pursuant to Contract DE-FC05-09CE-40905, awarded by the U.S. Department of Energy.

FIELD AND BACKGROUND OF THE INVENTION

The present invention relates, in general, to the pulp and paper industry, and in particular, to a new and useful apparatus, system and method for on-line, in-situ monitoring and/or controlling the concentration of lignin in wood pulp and black liquor.

In the pulp and paper industry, the production of paper products requires that lignin be partially removed from the wood chip feed stock prior to making paper products. Lignin is a polymer of complex chemical structure which "cements" the wood's cellulose fibers together. The process by which lignin is removed is referred to as delignification. The most prevalent method of delignification is by chemical means in which raw wood chips and chemicals are combined at controlled pressure and temperature in a vessel known as a digester. While in the digester, the amount of lignin removed from the wood chips determines the product quality, the product yield, the amount of energy consumed, the quantity of chemicals consumed, and the product cost. Fluid drained from the digester during delignification contains lignin removed from the wood chips and is referred to as "black liquor". The black liquor is used as fuel to the boiler to produce process steam.

The measurement of the residual lignin remaining in the pulp, which exits the digester, is most commonly carried out by laboratory analysis of hourly samples of the digester output (samples are typically obtained at the last stage of the brownstock washer). The lab analysis takes approximately one hour and therefore is a poor method for providing process control feedback and cannot be used for feedforward control. This lab analysis is a back titration method which measures the consumption of potassium permanganate and is only an approximation to the lignin concentration. The output of the titration analysis is referred to as a "KAPPA Number" and the procedure is documented in TAPPI procedure T236 cm-85, "KAPPA Number of Pulp". A number of manufacturers have produced automatic sampling and titration devices which have been tried in pulp mill situations but they have been mostly unsuccessful in providing accurate long-term results and did not eliminate the hour delay between the process and measurement of residual lignin.

The ultraviolet absorption and fluorescence properties of lignin have long been known and a number of researchers have reported results of measurements in solutions containing lignin. Both the absorption techniques (e.g. see Kleinert, T. N. and Joyce, C. S., "Short Wavelength Ultraviolet Absorption of Various Lignins and Related Substances," Part I, *Pulp and Paper Magazine Can.* 58, No. 5, April 1957, pp. 154–158.; Joyce, C. S. and Kleinert, T. N., Ibid., Part II, *Pulp and Paper Magazine Can.* 58, No. 6, May 1957, pp. 131–148.; Kleinert T. N. and Joyce, C. S., Ibid., Part III, *Pulp and Paper Magazine Can.* 58, No. 7, June 1957, pp. 215–219; Kleinert T. N. and Joyce, C. S., Ibid., Part IV, *Pulp and Paper Magazine Can.* 58, October 1957, pp 147–152; Hartler, N. and Norrstrom, H., "Light Absorbing Properties of Pulp and Pulp Components," *TAPPI Journal*, Vol. 52, No. 9, September 1969; Norrstrom, B. and Teder, A., "Absorption Bands in Electronic Spectra of Lignins, Part 2, Band Intensities for Alkali Lignins from Spruce," *Svensk Papperstidning*, 15 Jun. 1971; and Sjostrom, E. and Haglund, P., "Spectrophotometric Determination of the Dissolution of Lignin During Sulfite Cooling," TAPPI Journal, Vol. 47, No. 5, May 1964, pp 286–291) and the fluorescence techniques (e.g. see Bublitz, W. J., "Fluorescence of Pulping Liquors: A Tool for Digester Control?", pp 73–76; Baumgartner, D. J., Feldman, M. H., and Gibbons, C. L., "A Procedure for Tracing Kraft Mill Effluent From an Ocean by Constituent Fluorescence," *Water Research*, Pergamom Press, Vol. 4, 1971, pp 533–544; Bublitz, W. J. and Wade, D. C., "Applied Waste Liquor Fluorescence to Control Pulp Quality," *Svensk Paploerstidnin*, No. 18, 1979, pp 535–538; Wilander, A., Kvarnas, H. and Lindell, T., "A Modified Fluorometric Method for Measurement of Lignin Sulfonates and Its In-Situ Application in Natural Waters," Water Research, Vol. 8, 1974, pp 1037–1045; and Demas, J. N., *Excited State Lifetime Measurements*, Academic Press, New York, 1983, pp 53–58), have all been applied to very dilute solutions.

The fluorescence techniques have been used primarily as a method of detecting trace quantities in effluent streams. All of these approaches made use of very dilute lignin solutions where the absorption and fluorescence signal are linearly related to lignin concentration. The concentration of lignin in these solutions is typically 2000–10,000 times more dilute than the concentration of lignin in "black liquor" found in the pulping process. Thus, use of these techniques requires precise sample preparation prior to measurement. A number of devices which attempt to monitor the lignin concentration in "black liquor" during the pulping process by UV absorption techniques (alone or in combination with chemical analysis) have been produced. See Tikka, P. O., and Virkoka, N. E., "A New Kraft Pulping Analyzer for Monitoring Organic and Inorganic Substances," *TAPPI Journal*, June 1966, pp 66–71; Williams, D. J., "The Application of Ultra-Violet Absorption Characteristic of Lignin to the Control of Pulp Uniformity," *Appita*, Vol. 22, No. 2, September 1968, pp 45–52; and Capart, R., Obese-Jecty, K., Le Cardinal, G., and Gelus, M., "Contribution to the On-Line Kraft Pulping Control," *PRP* 4 *Proceedings*, Ghent, 1980, pp 121–128. These devices require sample preparation and dilution prior to measurement and are therefore not in-situ, not real-time, and introduce sampling and dilution errors.

Use of ultraviolet absorption has recently been extended to the measurement of residual lignin in wood pulp (see Kubulnieks, E., Lundqvist, S., and Pettersson, T., "The STFI OPTI-Kappa Analyzer, Applications and Accuracy," *TAPPI Journal*, November 1987, pp 38–42). This device is marketed by Asea Brown Boveri under the trade name "Opti-Kappa Analyzer". In this approach, the pulp stream is sampled approximately once every 5 minutes. The pulp sample is screened, washed thoroughly, and diluted significantly. The diluted sample is circulated in a loop where UV light absorption is measured over a prescribed time period and the pulp concentration in the slurry, i.e. pulp consistency, is measured independently. This system involves sampling error, screening error, and pulp consistency measurement error. Although the system provides results much faster than the conventional lab titration process, it is still off-line. The washing requirements of this device are stringent since any small amount of black liquor remaining in the diluted solution will absorb UV light and produce error. Bannier Technology Group (BTG, Inc.) also offers a device

which operates on a similar principle but uses UV reflection rather than absorption. The BTG device is marketed under the name "KNA-5000 Kappa Number Analyzer".

All of the investigations and devices discussed so far used broadband lamps as the source of UV light. In 1986, researchers at the National Bureau of Standards (see Horvath, J. J., Semerjian, H. G., "Laser Excited Fluorescence Studies of Black Liquor," *Proceedings of the SPIE*, Vol. 665, June 1986, pp 258–264) performed fluorescence tests on diluted black liquor samples using a laser as the source of UV light. Although their investigation resulted in better signal to noise ratios, they essentially did not extend the art beyond that of previous investigators. They were only able to obtain a functional relationship between fluorescence and lignin concentration in very dilute samples of black liquor (less than 1300 ppm, which is orders of magnitude less than the in-situ concentrations) and did not investigate pulp at all. They did not provide any insight into how one might be able to use either UV absorption or fluorescence techniques to extend the useful measurement range beyond the highly diluted state.

They did mention that this process was a candidate for in-situ monitoring but provided no rational explanation of how the dilution requirement could be overcome. They did mention that the measurement could be made more acceptable for field use by using optical fibers to guide the UV excitation light to the process stream and carry the fluorescence signal back to the opto-electronics unit.

SUMMARY OF THE INVENTION

Based on a desire to meet the need for an on-line, real-time device which could monitor the concentration of lignin in wood pulp and black liquor, the present invention resulted from a project which examined the fluorescence of black liquor and wood pulp under excitation by various narrowband wavelengths of UV light. It is believed that these wood pulp experiments were the first ever performed and that the results are novel in that a completely unexpected phenomenon was discovered. Namely, when the concentration of lignin in the specimen is increased beyond the very dilute regime, which had been studied earlier by others, the fluorescence signal intensity levels off and then begins to decrease with increasing concentrations of lignin. The region of most interest to on-line pulping is represented by a monotonically decreasing function of fluorescence vs. lignin concentration.

These earlier experiments led to three technical enhancements which improve accuracy and resolution of the measurement of lignin in an undiluted product. These three enhancements are:

1. Use of various single UV wavelengths to discriminate between the fluorescence of lignin and any potential interferents.
2. Use of time resolved fluorescence to eliminate fluorescence from non-lignins and to make the functional relationship between fluorescence and lignin concentration even more steep, thus resulting in improved resolution in highly concentrated substances.
3. Use of phase resolved fluorescence to eliminate the fluorescence from non-lignins.

U.S. Pat. Nos. 5,220,172 and 5,216,483 are based on these ideas.

It is an object of the present invention to provide an on-line measurement of lignin in wood pulp by employing a color shift of fluorescence. Accordingly, one aspect of the present invention is drawn to an apparatus for measuring lignin concentration in at least one undiluted sample on a real-time, in-situ basis. The apparatus comprises light source means for applying excitation light at a selected wavelength to the at least one undiluted sample to produce fluorescent emission light having a spectral distribution of fluorescent intensity. Light detector means are provided for detecting the fluorescent intensity of the emission light and determining the spectral distribution of the fluorescent intensity and establishing signals indicative thereof. Signal processing means are operatively connected to the light detector means for calculating lignin concentration from the spectral distribution signal and producing a signal indicative thereof. Finally, means are provided for displaying the signal indicative of the calculated lignin concentration for viewing by an operator.

Another aspect of the present invention is drawn to a system for controlling a delignification process by measuring lignin concentration in at least one undiluted sample in the process on a real-time, in-situ basis to produce a signal indicative of lignin concentration in the sample. The system comprises light source means for applying excitation light at a selected wavelength to the at least one undiluted sample to produce fluorescent emission light having a spectral distribution of fluorescent intensity. Light detector means are provided for detecting the fluorescent intensity of the emission light and determining the spectral distribution of the fluorescent intensity and establishing signals indicative thereof. Signal processing means are operatively connected to the light detector means for calculating lignin concentration from the spectral distribution signal and producing a signal indicative thereof. Finally, process control elements are provided and connected to the signal processing means for controlling the delignification process in response to the lignin concentration signal.

Yet another aspect of the present invention is drawn to a method for controlling a delignification process by measuring lignin concentration in at least one undiluted sample on a real-time, in-situ basis. The steps of this method comprise: applying an excitation light at a selected wavelength to the at least one undiluted sample to cause the sample to emit fluorescent emission light; determining a spectral distribution of the fluorescent emission light and establishing signals indicative thereof; calculating the lignin concentration in the sample from the spectral distribution signals; and controlling the delignification process by providing the signal indicative of the calculated lignin concentration to process control elements associated with the delignification process and responsive to said lignin concentration signal.

The various features of novelty which characterize the invention are pointed out with particularity in the claims annexed to and forming a part of this disclosure. For a better understanding of the invention, its operating advantages and specific objects attained by its uses, reference is made to the accompanying drawings and descriptive matter in which preferred embodiments of the invention are illustrated.

BRIEF DESCRIPTION OF THE DRAWINGS

In the drawings:

FIG. 1 is a graph plotting fluorescence spectra for pulp samples with three different Kappa numbers;

FIG. 2 is a graph plotting wavelength centroid versus Kappa number for pulp samples;

FIG. 3 is a graph plotting a ratio of fluorescence intensity over two emission wavelength ranges versus Kappa numbers;

FIG. 4 is a schematic block diagram of a system employing the lignin concentration apparatus according to the present invention;

FIG. 5 is schematic block diagram of a system using a wavelength centroid method according to the present invention; and

FIG. 6 is a schematic block diagram of a system using a band-ratio method according to the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

When light is absorbed, the absorber is raised to an energy level above the ground state. In most materials, the excess energy is quickly shared via collisions among the molecules in the vicinity of the absorption event resulting in a rise in the temperature. In some cases, however, a portion of the excess energy is given off in the form of a radiated photon. The excitation/emission process is referred to as fluorescence and has been widely used as an analytical technique.

Analytic capability is based on the fact that the spectral distribution of the fluorescent emission, $F(\lambda)d(\lambda)$ (where λ is the wavelength) is characteristic of the fluorescing material while the total fluorescence, F, defined by

$$F \equiv \int_0^\infty F(\lambda)d\lambda$$

is dependent on the concentration of the fluorescing material.

The approaches described in U.S. Pat. Nos. 5,220,172 and 5,216,483 involve the measurement of the total fluorescence, F. The present invention is based upon using the spectral distribution of the intensity of the fluorescent emission light as a measure of lignin concentration.

Fluorescence spectra from all pulp samples in a test series look superficially very much alike, as illustrated in FIG. 1. When normalized to the same height and overlaid for comparison, however, the three curves, derived from three different Kappa numbers, are slightly offset from one another.

In order to quantify this offset shift, the wavelength centroid, λ cent, of the spectral emission is calculated by the equation:

$$\lambda\text{ cent} \equiv \frac{\int_0^\infty \lambda F(\lambda)d\lambda}{\int_0^\infty F(\lambda)d\lambda}$$

FIG. 2 plots the wavelength centroid, λ cent, as a function of the Kappa number of the pulp samples. The Kappa number is the standard measure of the lignin concentration.

As an alternative measure of the spectral shift, the present invention utilizes the ratio, R, of the fluorescence integrated over two wavelength ranges, one comprised of longer wavelengths and the other comprised of shorter wavelengths. Specifically for the data shown in FIG. 3, this is determined by:

$$R \equiv \frac{\int_{\lambda=509}^{\lambda=564} F(\lambda)d\lambda}{\int_{\lambda=371}^{\lambda=427} F(\lambda)d\lambda}$$

where the limits on the integrals are given in nm.

FIG. 3 shows that this ratio R provides much the same measure of the offset shift as does the wavelength centroid, λ cent. Although both approaches provide essentially equivalent results, the hardware required to implement these two approaches is quite different as is described below.

FIG. 4 is a schematic block diagram of the major components of the present invention. Excitation light from a light source **10** is delivered to an undiluted pulp or liquid sample **20** of interest by excitation optics **30** where it excites the fluorescence of the pulp or liquid sample **20**. The pulp or liquid sample **20** is typically being conveyed through a pipe or conduit **25** forming a portion of the overall plant delignification system; alternatively pulp or liquid sample **20** could comprise a bypass line which conveys a portion of the total pulp or liquid flow taken from such a pipe or conduit **25**. In either case, a portion of the fluorescent emission light is gathered by the collection optics **40** and delivered to a detector/analyzer **50**. A signal processor **60** uses the output from detector/analyzer **50** to produce a calculated lignin concentration signal **65** representative of the calculated lignin concentration in the pulp or liquid sample **20**.

The calculated lignin concentration signal **65** can be used in a variety of ways. First, lignin concentration signal **65** can be displayed via display means **66** for viewing by a human operator concerned with the delignification process being monitored. Display means **66** can take any known analog and/or digital form. Second, lignin concentration signal **65** can be sent to one or more process control elements, schematically represented at **69**, for use in controlling the pulping/delignification process via known feedforward and/or feedback control elements. These control elements, as known to those skilled in the art, can use other signals **68** representative of other process parameters for the delignification process to control various aspects of the delignification process such as, by way of example and not limitation, the time of cooking of the pulp, the temperature during the process, flow rates of various substances to and from the process, and other controllable factors that affect the composition of the lignin. Indeed, a primary use envisioned for the lignin concentration signal **65** is to provide a continuous, real-time signal for use in feedback control of the paper pulping process and/or for feedforward control of the paper bleaching process. Finally, lignin concentration signal **65** can be sent to recording means, schematically represented at **67** and also of known construction, to produce a time history of the process which, together with signals **68** representative of other process parameters for the delignification process, could be used for later review and analysis. The present invention's ability to produce on-line, in-situ measurements can thus provide new insights into the delignification process and how varying controllable parameters can be used to optimize performance.

A pulsed Nitrogen laser having an output of 337 nm was used as an excitation source for the experimental data shown in FIGS. 2 and 3. This is a convenient, and perhaps preferred source, but the present invention does not require that the source **10** be pulsed, operate at 337 nm, or even be a laser.

In a preferred embodiment, both the excitation optics **30** and the collection optics **40** use optical fibers to allow

measurement at points remote from the source 10 and analyzer 50. Again, however, optical fiber based optics are not essential to the operation of the present invention.

The data illustrated in FIGS. 2 and 3 was taken from undiluted pulp samples in conditions simulating those at the last stage of a brownstock washer. This measurement position was chosen for initial consideration because it is where the samples for laboratory analyses are currently taken. There is, however, good reason to consider measurement at other positions in the process as well.

The two methods utilized by the present invention, i.e. (1) the wavelength centroid method and (2) the band-ratio method, utilize different analyzer/detector assemblies. Hardware for the wavelength centroid method is shown in FIG. 5. FIG. 5 shows the system employing fiber optic assemblies 70, 80 and 90 in order to provide measurement at several remote locations A, B . . . N. A single light source 10 provides excitation light along fibers 70 to probe assemblies 80 where it exits the fiber and illuminates the undiluted pulp or liquid sample 20 to be measured. The resultant fluorescent light from the pulp or liquid sample 20 is collected by probe assembly optics 80 and input to return fibers 90 which carry it to an analyzer/detector 50*a*.

In FIG. 5, the analyzer/detector 50*a* comprises a polychromator 100 for dispersing the light into a spectrum. Polychromator 100 is operatively connected to a two-dimensional charge-coupled device (CCD) array detector 110 that can simultaneously but separately detect the spectrum from each of the fibers 90. An analog-to-digital A/D converter board 120 is operatively connected to the detector 110 in order to digitize the CCD signals. Signal processing is provided by a computer 130, which is operatively connected to the A/D converter 120, and also controls the detector readout.

The output provided by the computer 130 is the lignin concentration at each of the measurement points A, B . . . . N determined by calculating the wavelength centroid of the light from each probe assembly 80 according to the wavelength centroid method described above. The calculated lignin concentration signal 133 can be displayed on a display 135 for reading by an operator, or the signal can be sent to process control elements schematically represented at 140 for use in controlling the pulping/delignification process via known feedforward and/or feedback control elements. These control elements, as known to those skilled in the art, control various aspects of the delignification process such as, by way of example and not limitation, the time of cooking of the pulp, the temperature during the process, flow rates to and from the process, and other controllable factors that affect the composition of the lignin. Additionally, the calculated lignin concentration signal 133 can be sent to recording means, schematically represented at 145 and of known construction, to produce a time history of the process which, together with signals 150 representative of other process parameters for the delignification process, could be used for later review and analysis.

The hardware used for implementing the band-ratio method according to the present invention is shown in FIG. 6. The light source 10 and fiber optic assemblies and probes 70, 80 and 90 are the same devices used for the wavelength centroid method of FIG. 5. In FIG. 6, an analyzer/detector section 50*b* comprises a dichroic filter or beam splitter 155 for each measurement point A, B, ... N which is operatively connected to pick-up fibers 90 in order to separate the fluorescent light into two desired bands, one comprised of longer wavelengths and the other comprised of shorter wavelengths. A pair of detectors 158 near each dichroic filter or beam splitter 155, for each measurement point, provides a signal proportional to the total fluorescence passed by the dichroic filters or beam splitters 155. Lines 159 provide these signals to a signal processor 160 which comprises circuitry to determine and convert the ratio of the signals from the two wavelength bands to a Kappa number which is output at display means 165, using either analog or digital signal processing techniques. Again, the signal 168 representative of the calculated lignin concentration can be displayed on a display 165 for reading by an operator, or the signal 168 can be sent to process control elements schematically represented at 170 for use in controlling the pulping/delignification process via known feedforward and/or feedback control elements. These control elements, as known to those skilled in the art, again control various aspects of the delignification process such as, by way of example and not limitation, the time of making of the pulp, the temperature during the process, flow rates to and from the process, and other controllable factors that affect the composition of the lignin. Additionally, the calculated lignin concentration signal 168 can again be sent to recording means, schematically represented at 175 and of known construction, to produce a time history of the process which, together with signals 180 representative of other process parameters for the delignification process, could be used for later review and analysis.

For the embodiments shown in FIGS. 4–6, the signal processor 60, the computer 130, and the signal processing electronics 160 would typically measure lignin concentration on a real-time, in-situ basis over a defined time interval. The light sources in each embodiment would apply excitation light at selected wavelengths, for a specified duration, and over the defined time interval. A plurality of signals representative of plural lignin concentration measurements would then be obtained and which could be averaged together to obtain a calculated bulk lignin concentration signal that is representative of the lignin concentration in the bulk material being measured during the defined time interval. Of course, each of these aspects would be selected to suit a given application.

The present invention provides a sensor capable of measuring the lignin concentration in wood pulp at a pulp mill in real-time and in-situ, without the need for sample taking, sample preparation, and sample washing. The systems of the present invention, because they do not require sampling and preparation and because of the "discrimination" enhancements described above, can also be made to be more accurate than other available lignin sensor technologies. Because the systems according to the present invention require no dilution, potential errors due to dilution are eliminated.

The hardware employed by the present invention is easily joined to known fiber optic technology in order to permit hardened probes for single or multiple point sensing of pulp streams. This provides two major advantages. First, the source and analyzer can be located remotely and protected from the harsh environment. Second, a single source/analyzer package can be used via multiplexing to monitor several points in the process stream.

As an alternative to the multiplexing scheme described above, a time share multiplexing scheme could be employed. In this approach, the excitation light would be moved sequentially, such as by drive means schematically represented in FIGS. 5 and 6 at 200, from one delivery fiber to the next, interrogating the various measurement locations in sequence.

The present invention offers additional significant advantages related to the fact that the measurement is based on the

relative spectral distribution of the fluorescent light intensity, rather than the absolute magnitude of the fluorescence. Because of this approach, no compensation is required for variations in the excitation source output intensity, variations in the optical efficiency due to alignment changes, fiber transmission loss, etc., or variations due to contamination of the exposed optical surfaces of the probe due to dust, splashes, etc.

The present invention can be applied for excitation by any source that provides measurable fluorescent intensity whether it be a laser or not, and whether it be pulsed or continuous, or by direct illumination or illumination remotely through an optical fiber. The present invention is useful for analysis of dry, undiluted pulp samples or pulp slurries of various consistencies, wood pulp or other process samples that fluoresce, such as petroleum products, food stuffs, pharmaceuticals, etc., and in the application of other methods for quantifying a color shift such as the difference between the intensity of two wavelength bands.

While specific embodiments of the invention have been shown and described in detail to illustrate the application of the principles of the invention, it will be understood that the invention may be embodied otherwise without departing from such principles.

We claim:

1. An apparatus employing a color shift of fluorescence for measuring lignin concentration in at least one undiluted sample on a real-time, in-situ basis based upon a spectral distribution of fluorescent emission light from the sample, comprising:

light source means for applying excitation light at a selected wavelength to the at least one undiluted sample for causing the sample to produce fluorescent emission light having a spectral distribution of fluorescent intensity;

light detector means for detecting the fluorescent intensity of the emission light and determining the spectral distribution of fluorescent intensity and establishing signals indicative thereof;

signal processing means operatively connected to the light detector means for calculating a lignin concentration from the spectral distribution signals and producing a signal indicative thereof; and

means for displaying the signal indicative of the calculated lignin concentration for viewing by an operator.

2. The apparatus according to claim 1, comprising probe means, connected to the light source means and the light detector means, located at the at least one sample for conveying the excitation light to the at least one sample and conveying the fluorescent emission light to the light detector means.

3. The apparatus according to claim 1, wherein the light detector means comprises polychromator means for dispersing the fluorescent intensity of the emission light into a spectrum.

4. The apparatus according to claim 3, wherein the light detector means further comprises array detector means operatively connected to the polychromator means for simultaneously and separately detecting the spectrum of a plurality of samples.

5. The apparatus according to claim 4, wherein the light detector means further comprises analog-to-digital converter means operatively connected to the array detector means for converting the spectral distribution signals into a digital signal.

6. The apparatus according to claim 4, wherein the array detector means comprises a two-dimensional charge-coupled device (CCD) array detector.

7. The apparatus according to claim 5, wherein the signal processing means comprises control means for controlling the polychromator means and the array detector means and for outputting the digital signal.

8. The apparatus according to claim 7, comprising a plurality of samples.

9. The apparatus according to claim 8, comprising probe means connected to the light source means and the polychromator means and located at the samples for conveying the excitation light to the samples and for conveying the fluorescent emission light of the samples to the polychromator means.

10. The apparatus according to claim 1, wherein the light detector means comprises filter means for separating the fluorescent intensity of the emission light into two bands.

11. The apparatus according to claim 10, wherein the light detector means further comprises detecting means for detecting each band and conveying a signal proportional to the fluorescent intensity of each band of the emission light.

12. The apparatus according to claim 11, wherein the signal processing means converts the proportional signals into a Kappa number.

13. The apparatus according to claim 12, comprising a plurality of samples.

14. The apparatus according to claim 13, comprising probe means connected to the light source means and the filter means and located at the samples for conveying the excitation light to the samples and for conveying the fluorescent emission light from the samples to the filter means.

15. The apparatus according to claim 1, wherein the light source means is a laser.

16. The apparatus according to claim 1, wherein the light source means is a pulsed laser.

17. The apparatus according to claim 16, wherein the light source means is a pulsed nitrogen laser.

18. The apparatus according to claim 17, wherein the light source means is a pulsed nitrogen laser having an output of 337 nm.

19. The apparatus according to claim 1, comprising:

means for calculating the lignin concentration from the spectral distribution signals a plurality of times over a defined time interval to produce a plurality of lignin concentration signals; and

means for processing said plurality of lignin concentration signals to produce a calculated bulk lignin concentration signal that is representative of the lignin concentration in the sample during the defined time interval.

20. A system for controlling a delignification process which employs a color shift of fluorescence to measure lignin concentration in at least one undiluted lignin sample in the process on a real-time, in-situ basis to produce a signal indicative of lignin concentration in the sample based upon a spectral distribution of fluorescent emission light from the sample, comprising:

light source means for applying excitation light at a selected wavelength to the at least one undiluted sample for causing the sample to produce fluorescent emission light having a spectral distribution of fluorescent intensity;

light detector means for detecting the fluorescent intensity of the emission light and determining the spectral distribution of fluorescent intensity and establishing signals indicative thereof;

signal processing means operatively connected to the light detector means for calculating lignin concentration from the spectral distribution signals and producing a signal indicative thereof; and

process control elements, connected to the signal processing means, for controlling the delignification process in response to the lignin concentration signal.

21. The system according to claim 20, comprising recording means for recording the variation with time of said lignin concentration signal and other process parameters associated with the delignification process, to produce a time history of these process parameters.

22. A method for controlling a delignification process which employs a color shift of fluorescence to measure lignin concentration in at least one undiluted sample in the process on a real-time, in-situ basis to produce a signal indicative of lignin concentration in the sample based upon a spectral distribution of fluorescent emission light from the sample, the method comprising the steps of:

applying an excitation light at a selected wavelength to the at least one undiluted sample to cause the sample to emit fluorescent emission light;

determining a spectral distribution of the fluorescent emission light and establishing signals indicative thereof;

calculating the lignin concentration from the spectral distribution signals; and

controlling the delignification process by providing the signal indicative of the calculated lignin concentration to process control elements associated with the delignification process and responsive to said lignin concentration signal.

23. The method according to claim 22, further comprising the steps of:

calculating the lignin concentration from the spectral distribution signals a plurality of times over a defined time interval to produce a plurality of lignin concentration signals; and

processing said plurality of lignin concentration signals to produce a calculated bulk lignin concentration signal that is representative of the lignin concentration in the sample during the defined time interval.

* * * * *

NOTICE OF OFFICE PLAN TO CEASE SUPPLYING COPIES OF CITED U.S. PATENT REFERENCES WITH OFFICE ACTIONS, AND PILOT TO EVALUATE THE ALTERNATIVE OF PROVIDING ELECTRONIC ACCESS TO SUCH U.S. PATENT REFERENCES

Summary

The United States Patent and Trademark Office (Office or USPTO) plans in the near future to: (1) cease mailing copies of U.S. patents and U.S. patent application publications (US patent references) with Office actions except for citations made during the international stage of an international application under the Patent Cooperation Treaty and those made during reexamination proceedings; and (2) provide electronic access to, with convenient downloading capability of, the US patent references cited in an Office action via the Office's private Patent Application Information Retrieval (PAIR) system which has a new feature called "E-Patent Reference." Before ceasing to provide copies of U.S. patent references with Office actions, the Office shall test the feasibility of the E-Patent Reference feature by conducting a two-month pilot project starting with Office actions mailed after December 1, 2003. The Office shall evaluate the pilot project and publish the results in a notice which will be posted on the Office's web site (www.USPTO.gov) and in the Patent Official Gazette (O.G.). In order to use the new E-Patent Reference feature during the pilot period, or when the Office ceases to send copies of U.S. patent references with Office actions, the applicant must: (1) obtain a digital certificate from the Office; (2) obtain a customer number from the Office, and (3) properly associate applications with the customer number. The pilot project does not involve or affect the current Office practice of supplying paper copies of foreign patent documents and non-patent literature with Office actions. Paper copies of references will continue to be provided by the USPTO for searches and written opinions prepared by the USPTO for international applications during the international stage and for reexamination proceedings.

Description of Pilot Project to Provide Electronic Access to Cited U.S. Patent References

On December 1, 2003, the Office will make available a new feature, E-Patent Reference, in the Office's private PAIR system, to allow more convenient downloading of U.S. patents and U.S. patent application publications. The new feature will allow an authorized user of private PAIR to download some or all of the U.S. patents and U.S. patent application publications cited by an examiner on form PTO-892 in Office actions, as well as U.S. patents and U.S. patent application publications submitted by applicants on form PTO/SB08 (1449) as part of an IDS. The retrieval of some or all of the documents may be performed in one downloading step with the documents encoded as Adobe Portable Document format (.pdf) files, which is an improvement over the current page-by-page retrieval capability from other USPTO systems.

Steps to Use the New E-Patent Reference Feature During the Pilot Project and Thereafter

Access to private PAIR is required to utilize E-Patent Reference. If you don't already have access to private PAIR, the Office urges practitioners, and applicants not represented by a practitioner, to take advantage of the transition period to obtain a no-cost USPTO Public Key Infrastructure (PKI) digital certificate, obtain a USPTO customer number, associate all of their pending and new application filings with their customer number, install no-cost software (supplied by the Office) required to access private PAIR and E-Patent Reference feature, and make appropriate arrangements for Internet access. The full instructions for obtaining a PKI digital certificate are available at the Office's Electronic Business Center (EBC) web page at: <http://www.uspto.gov/ebc/downloads.html>. Note that a notarized signature will be required to obtain a digital certificate.

To get a Customer Number, download and complete the Customer Number Request form, PTO-SB125, at: http://www.uspto.gov/web/forms/sb0125.pdf. The completed form can then be transmitted by facsimile to the Electronic Business Center at (703) 308-2840, or mailed to the address on the form. If you are a registered attorney or patent agent, then your registration number must be associated with your customer number. This is accomplished by adding your registration number to the Customer Number Request form. A description of associating a customer number with an application is described at the EBC web page at: http://www.uspto.gov/ebc/registration_pair.html.

The E-Patent Reference feature will be accessed using a new button on the private PAIR screen. Ordinarily all of the cited U.S. patent and U.S. patent application publication references will be available over the Internet using the Office's new E-Patent Reference feature. The size of the references to be downloaded will be displayed by E-Patent Reference so the download time can be estimated. Applicants and registered practitioners can select to download all of the references or any combination of cited references. Selected references will be downloaded as complete documents as Adobe Portable Document Format (.pdf) files. For a limited period of time, the USPTO will include a copy of this notice with Office actions to encourage applicants to use this new feature and, if needed, to take the steps outlined above in order to be able to utilize this new feature during the pilot and thereafter.

During the two-month pilot, the Office will evaluate the stability and capacity of the E-Patent Reference feature to reliably provide electronic access to cited U.S. patent and U.S. patent application publication references. While copies of U.S. patent and U.S. patent application publication references cited by examiners will continue to be mailed with Office actions during the pilot project, applicants are encouraged to use the private PAIR and the E-Patent Reference feature to electronically access and download cited U.S. patent and U.S. patent application publication references so the Office will be able to objectively evaluate its performance. The public is encouraged to submit comments to the Office on the usability and performance of the E-Patent Reference feature during the pilot. Further, during the pilot period registered practitioners, and applicants not represented by a practitioner, are encouraged to experiment with the feature, develop a proficiency in using the feature, and establish new internal processes for using the new access to the cited U.S. patents and U.S. patent application publications to prepare for the anticipated cessation of the current Office practice of supplying copies of such cited

references. The Office plans to continue to provide access to the E-Patent Reference feature during its evaluation of the pilot.

Comments

Comments concerning the E-Patent Reference feature should be in writing and directed to the Electronic Business Center (EBC) at the USPTO by electronic mail at eReference@uspto.gov or by facsimile to (703) 308-2840. Comments will be posted and made available for public inspection. To ensure that comments are considered in the evaluation of the pilot project, comments should be submitted in writing by January 15, 2004.

Comments with respect to specific applications should be sent to the Technology Centers' customer service centers. Comments concerning digital certificates, customer numbers, and associating customer numbers with applications should be sent to the Electronic Business Center (EBC) at the USPTO by facsimile at (703) 308-2840 or by e-mail at EBC@uspto.gov.

Implementation after Pilot

After the pilot, its evaluation, and publication of a subsequent notice as indicated above, the Office expects to implement its plan to cease mailing paper copies of U.S. patent references cited during examination of non provisional applications on or after February 2, 2004; although copies of cited foreign patent documents, as well as non-patent literature, will still be mailed to the applicant until such time as substantially all applications have been scanned into IFW.

For Further Information Contact

Technical information on the operation of the IFW system can be found on the USPTO website at http://www.uspto.gov/web/patents/ifw/index.html. Comments concerning the E-Patent Reference feature and questions concerning the operation of the PAIR system should be directed to the EBC at the USPTO at (866) 217-9197. The EBC may also be contacted by facsimile at (703) 308-2840 or by e-mail at EBC@uspto.gov.

Date: 12/1/03

Nicholas P. Godici

Nicholas P. Godici
Commissioner for Patents

REVISED AMENDMENT PRACTICE: 37 CFR 1.121 CHANGED
COMPLIANCE IS MANDATORY - Effective Date: July 30, 2003

All amendments filed on or after the effective date noted above must comply with revised 37 CFR 1.121. See Final Rule: **Changes To Implement Electronic Maintenance of Official Patent Application Records** (68 *Fed. Reg.* 38611 (June 30, 2003), posted on the Office's website at: http://www.uspto.gov/web/patents/ifw/ with related information. The amendment practice set forth in revised 37 CFR 1.121, and described below, replaces the voluntary revised amendment format available to applicants since February 2003. **NOTE: STRICT COMPLIANCE WITH THE REVISED 37 CFR 1.121 IS REQUIRED AS OF THE EFFECTIVE DATE (July 30, 2003).** The Office will notify applicants of amendments that are not accepted because they do not comply with revised 37 CFR 1.121 via a Notice of Non-Compliant Amendment. See MPEP 714.03 (Rev. 1, Feb. 2003). The non-compliant section(s) will have to be corrected and the entire corrected section(s) resubmitted within a set period.

Bold underlined italic font has been used below to highlight the major differences between the revised 37 CFR 1.121 and the voluntary revised amendment format that applicants could use since February, 2003.
Note: The amendment practice for **reissues** and **reexamination proceedings**, except for drawings, has not changed.

REVISED AMENDMENT PRACTICE

I. Begin each section of an amendment document on a separate sheet:
Each section of an amendment document (*e.g.*, Specification Amendments, Claim Amendments, Drawing Amendments, and Remarks) must begin on a separate sheet. Starting each separate section on a new page will facilitate the process of separately indexing and scanning each section of an amendment document for placement in an image file wrapper.

II. Two versions of amended part(s) no longer required:
37 CFR 1.121 has been revised to no longer require two versions (a clean version and a marked up version) of each replacement paragraph or section, or amended claim. Note, however, the requirements for a clean version and a marked up version for substitute specifications under 37 CFR 1.125 have been retained.

A) **Amendments to the claims:**
Each amendment document that includes a change to an existing claim, cancellation of a claim or submission of a new claim, **must include a complete listing** of all claims in the application. After each claim number in the listing, the status must be indicated in a parenthetical expression, and **the text of each pending claim** (with markings to show current changes) must be presented. The claims in the listing will replace all prior claims in the application.

(1) The current status of all of the claims in the application, including any previously canceled, not entered or withdrawn claims, must be given in a parenthetical expression following the claim number using only one of the following seven status identifiers: (original), (currently amended), (canceled), (withdrawn), (new), ***(previously presented) and (not entered).*** The text of all pending claims, ***including withdrawn claims,*** must be submitted each time any claim is amended. Canceled ***and not entered*** claims must be indicated by only the claim number and status, without presenting the text of the claims.

(2) The text of all claims being currently amended must be presented in the claim listing with markings to indicate the changes that have been made relative to the immediate prior version. The changes in any amended claim must be shown by underlining (for added matter) or strikethrough (for deleted matter) with 2 exceptions: (1) for ***deletion of five characters or fewer, double brackets may be used (e.g., [[eroor]]); and (2) if strikethrough cannot be easily perceived (e.g., deletion of the number "4" or certain punctuation marks), double brackets must be used (e.g., [[4]]). As an alternative to using double brackets, however, extra portions of text may be included before and after text being deleted, all in strikethrough, followed by including and underlining the extra text with the desired change*** (e.g., ~~number 4 as~~ *number 14 as*). An accompanying clean version is not required and should not be presented. Only claims of the status "currently amended," and "withdrawn" that are being amended, may include markings.

(3) The text of pending claims not being currently amended, ***including withdrawn claims,*** must be presented in the claim listing in clean version, *i.e.*, without any markings. Any claim text presented in clean version will constitute an assertion that it has not been changed relative to the immediate prior version except to omit markings that may have been present in the immediate prior version of the claims.

(4) A claim being canceled must be listed in the claim listing with the status identifier "canceled"; the text of the claim must not be presented. Providing an instruction to cancel is optional.
(5) Any claims added by amendment must be presented in the claim listing with the status identifier "(new)"; the text of the claim must not be underlined.
(6) All of the claims in the claim listing must be presented in ascending numerical order. Consecutive canceled, or not entered, claims may be aggregated into one statement (*e.g.*, Claims 1 – 5 (canceled)).

Example of listing of claims (use of the word "claim" before the claim number is optional):

Claims 1-5 (canceled)

Claim 6 (previously presented): A bucket with a handle.

Claim 7 (withdrawn): A handle comprising an elongated wire.

Claim 8 (withdrawn): The handle of claim 7 further comprising a plastic grip.

Claim 9 (currently amended): A bucket with a ~~green~~ blue handle.

Claim 10 (original): The bucket of claim 9 wherein the handle is made of wood.

Claim 11 (canceled)

Claim 12 (not entered)

Claim 13 (new): A bucket with plastic sides and bottom.

B) Amendments to the specification:
Amendments to the specification, including the abstract, must be made by presenting a replacement paragraph or section or abstract marked up to show changes made relative to the immediate prior version. An accompanying clean version is not required and should not be presented. Newly added paragraphs or sections, including a new abstract (instead of a replacement abstract), must not be underlined. A replacement or new abstract must be submitted on a separate sheet, 37 CFR 1.72. If a substitute specification is being submitted to incorporate extensive amendments, both a clean version (which will be entered) and a marked up version must be submitted as per 37 CFR 1.125.

The changes in any replacement paragraph or section, or substitute specification must be shown by underlining (for added matter) or strikethrough (for deleted matter) with 2 exceptions: (1) for ***deletion of five characters or fewer, double brackets may be used (e.g., [[eroor]]); and (2) if strikethrough cannot be easily perceived (e.g., deletion of the number "4" or certain punctuation marks), double brackets must be used (e.g., [[4]]). As an alternative to using double brackets, however, extra portions of text may be included before and after text being deleted, all in strikethrough, followed by including and underlining the extra text with the desired change*** (*e.g.*, ~~*number 4 as*~~ *number 14 as*)

C) Amendments to drawing figures:
Drawing changes must be made by presenting replacement figures which incorporate the desired changes and which comply with 37 CFR 1.84. An explanation of the changes made must be presented either in the drawing amendments, or remarks, section of the amendment, ***and may be accompanied by a marked-up copy of one or more of the figures being amended, with annotations.*** Any replacement drawing sheet must ***be identified in the top margin as "Replacement Sheet"*** and include all of the figures appearing on the immediate prior version of the sheet, even though only one figure may be amended. ***Any marked-up (annotated) copy showing changes must be labeled "Annotated Marked-up Drawings" and accompany the replacement sheet in the amendment (e.g., as an appendix).*** The figure or figure number of the amended drawing(s) must **not** be labeled as "amended." If the changes to the drawing figure(s) are not accepted by the examiner, applicant will be notified of any required corrective action in the next Office action. No further drawing submission will be required, unless applicant is notified.

Questions regarding the submission of amendments pursuant to the revised practice set forth in this flyer should be directed to: Elizabeth Dougherty or Gena Jones, Legal Advisors, or Joe Narcavage, Senior Special Projects Examiner, Office of Patent Legal Administration, by e-mail to patentpractice@uspto.gov or by phone at (703) 305-1616.